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SPOOR AND FISHER

REPUBLIC OF SOUTH AFRICA
PATENTS ACT, 1978
APPLICATION FOR A PATENT
AND ACKNOWLEDGEMENT OF RECEIPT
(Section 30 (1) - Regulation 22)

REPUBLIC OF SOUTH AFRICA
REVENUE FORM P.1.

REVENUE STAMPS OR REVENUE FRANKING
MACHINE IMPRESSION R * 3000

INKOMSTE
REPUBLIEK VAN SUID AFRIKA
HR 70

OFFICIAL DATE STAMP

The grant of a patent is hereby requested by the undermentioned applicant on the basis of the present application filed in duplicate.

OFFICIAL APPLICATION NO.

| | | |
|----|----|--------|
| 21 | 01 | 806992 |
|----|----|--------|

S & F REFERENCE

| |
|----------|
| JP/J 099 |
|----------|

FULL NAME(S) OF APPLICANT(S)

| | |
|----|--|
| 71 | JOHANNESBURG CONSOLIDATED INVESTMENT COMPANY LIMITED |
|----|--|

ADDRESS(ES) OF APPLICANT(S)

| | |
|--|--|
| | Consolidated Building, cnr. Fox and Harrison Streets, Johannesburg |
|--|--|

TITLE OF INVENTION

| | |
|----|---|
| 54 | "ORGANIC DIAMMONIUM AND RELATED COMPOUNDS AS SOLVENT EXTRACTANTS" |
|----|---|

PRIORITY IS CLAIMED AS SET OUT ON THE ACCOMPANYING FORM P.2.

~~THIS APPLICATION IS FOR A PATENT OF ADDITION TO PATENT APPLICATION NO.~~

| | | |
|----|----|--|
| 21 | 01 | |
|----|----|--|

~~THIS APPLICATION IS A FRESH APPLICATION IN TERMS OF SECTION 27 AND BASED ON APPLICATION NO.~~

| | | |
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| 21 | 01 | |
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THIS APPLICATION IS ACCOMPANIED BY:

- ☒ 1. A single copy of a provisional or two copies of a complete specification of 23 pages.
- ☐ 2. Drawings of sheets
- ☒ 3. Publication particulars and abstract (Form P.8, in duplicate)
- ☐ 4. A copy of Figure of the drawings (if any) for the abstract.
- ☒ 5. Assignment of invention.
- ☐ 6. Certified priority document(s) (State number)
- ☐ 7. Translation of the priority document(s)
- ☐ 8. An assignment of priority rights
- ☒ 9. A copy of the Form P.2. and the specification of S.A. Patent Application No.

| | | |
|----|----|---------|
| 21 | 01 | 79/6097 |
|----|----|---------|
- ☐ 10. A declaration and power of attorney on Form P.3.
- ☐ 11. Request for ante-dating on Form P.4.
- ☐ 12. Request for classification on Form P.9.
- ☒ 13. Form P.2. in duplicate

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| 74 | ADDRESS FOR SERVICE: SPOOR AND FISHER, SANDTON, REPUBLIC OF SOUTH AFRICA |
|----|--|

Dated This 12th Day of NOVEMBER, 1980.

Spoor and Fisher
SPOOR AND FISHER
APPLICANTS PATENT ATTORNEYS

| |
|----------------------|
| RECEIVED |
| OFFICIAL DATE STAMP |
| REGISTRAR OF PATENTS |

REPUBLIC OF SOUTH AFRICA
PATENTS ACT, 1978
COMPLETE SPECIFICATION
(Section 30(1) - Regulation 28)

OFFICIAL APPLICATION NO.

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| 21 | 01 | 806992 |
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| 22 | NOVEMBER 12, 1980 |
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| 51 | B01D C07C C07D |
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FULL NAME(S) OF APPLICANT(S)

| | |
|----|--|
| 71 | JOHANNESBURG CONSOLIDATED INVESTMENT COMPANY LIMITED |
|----|--|

FULL NAME(S) OF INVENTOR(S)

| | |
|----|-------------------------------|
| 72 | JAN GYSBERT HERMANUS DU PREEZ |
|----|-------------------------------|

TITLE OF INVENTION

| | |
|----|---|
| 54 | "ORGANIC DIAMMONIUM AND RELATED COMPOUNDS AS SOLVENT EXTRACTANTS" |
|----|---|

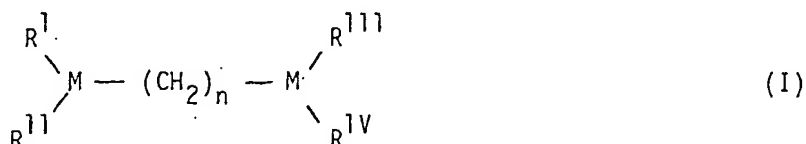
BACKGROUND TO THE INVENTION

THIS invention relates to solvent extraction and, in particular to the recovery of metal ions from aqueous solution by means of solvent extraction, and to novel compounds for use as extractants in such solvent extraction.

SUMMARY OF THE INVENTION

5 According to one aspect of the invention there is provided a compound selected from:

(a) a ditertiary amine, phosphine or arsine of the general formula



where M = N, P or As

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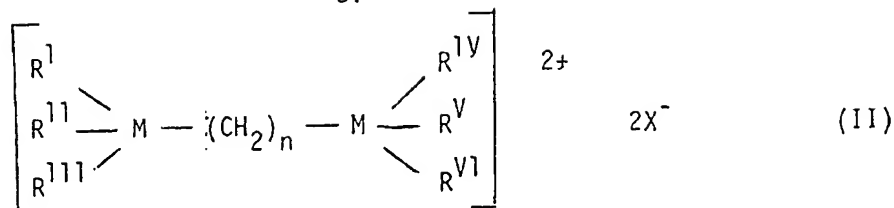
n = 2 to 10 inclusive

R^I , R^{II} , R^{III} , and R^{IV} are the same or different and each represents an alkyl or an aryl group having 7 to 12 carbon atoms; and

15

(b) a diquaternary ammonium, phosphonium or arsonium salt of the general formula

3.



where M = N, P or As

n = 2 to 10 inclusive

X = Cl⁻, Br⁻, I⁻, SCN⁻, NO₃⁻, HSO₄⁻ or SO₄⁼

R^I, R^{II}, R^{III}, R^{IV}, R^V, and R^{VI} are the same or different and

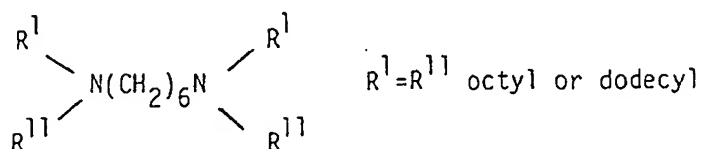
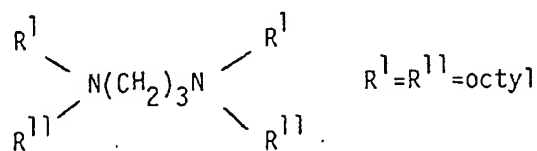
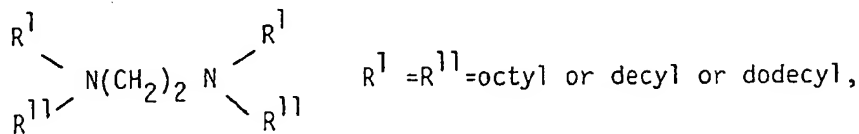
5 each represents an alkyl or an aryl group having 1 to 12 carbon atoms or H, provided that at least four of R^I, R^{II}, R^{III}, R^{IV}, R^V and R^{VI} represent an alkyl or an aryl group having 7 - 12 carbon atoms.

According to another aspect of the invention there is provided a compound of the formula (I) or (II) as defined above for use in the solvent extraction of metal ions from an aqueous solution containing the ions in the form of anionic complexes.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

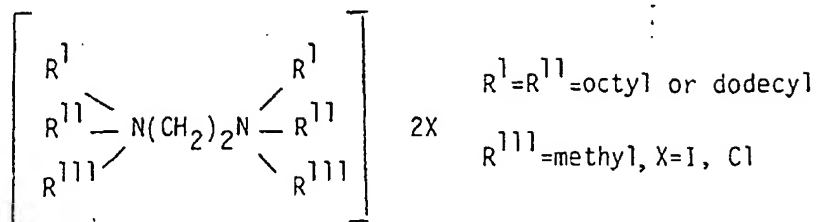
Preferred compounds of the invention are those in which the substituents on one N, P or As atom are the same as those on the other N, P or As
10 atom.

Particularly preferred compounds of the invention are ditertiary amine compounds of the formulae

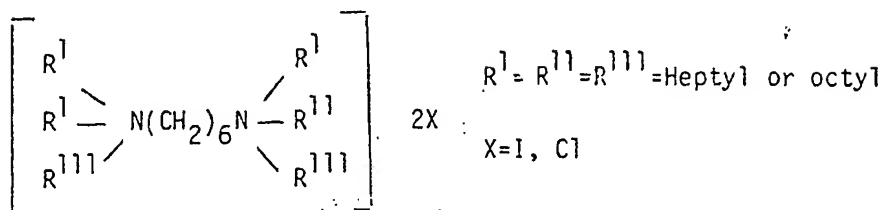


4.

and diquatery ammonium compounds of the formulae:



and



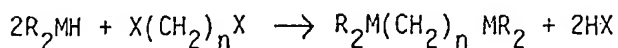
Examples of preferred compounds are
 N,N,N',N',-Tetraoctylethylenediamine (Tetocen)

- 5 N',N',N',N,N,N,-hexaoctylhexamethylene diammonium diiodide (Hexochem).
 N,N',-dimethyl-N,N,N',N',-tetraoctylethylene diammonium diiodide (Dimtetocen).

The compounds of this invention may be produced by several procedures.

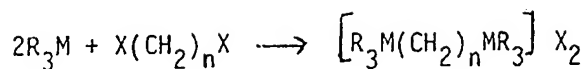
- 10 The compounds of the formula (1) can be prepared by the reaction shown in Equation A.

EQUATION A



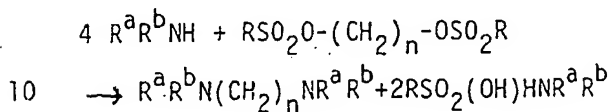
wherein R is an alkyl or aryl group having 7 to 12 carbon atoms,
 X is halogen, and M and n are as defined above.

- 15 Similarly, the compounds of the formula (11) can be prepared by the reaction shown in Equation B.

EQUATION B

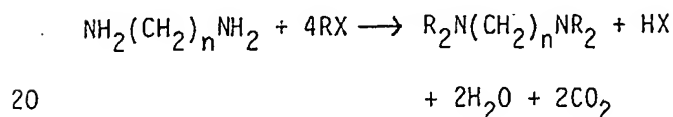
wherein R is an alkyl or aryl group having 7 to 12 carbon atoms and M, X and n are as defined above.

- 5 The compounds of the formula (1) in which M = N can be prepared by the reaction of a dialkylamine with glycol disulphonate according to Equation C.

EQUATION C

wherein R^a, R^b and R may be the same or different and each represents an alkyl or an aryl group having 7 to 12 carbon atoms and n is as defined above.

- 15 A novel method of preparing the compounds of formula (1) in which M = N involves the direct alkylation of a primary 1,n diamino alkane (n = 2, ..., 10) in a suitable alcoholic solvent mixture in the presence of an inorganic base according to Equation D.

EQUATION D

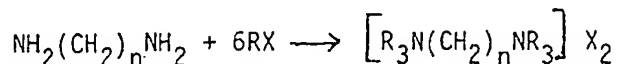
wherein R represents an alkyl or an aryl group having 7 to 12 carbon atoms,

X represents a halogen and n is as defined above.

- 25 The preferred alcoholic solvent mixture is a mixture of ethanol and hexane, preferably in the ratio ethanol : 40/hexane : 60.
The preferred inorganic base is potassium carbonate.

In a similar manner the compounds of the formula (11) in which $M = N$ can be prepared according to Equation E.

EQUATION E



5 wherein R, n and X are as defined above.

The preferred alcoholic solvent mixture is a mixture of ethanol and DMF (N,N'-dimethyl formamide), preferably in the ratio DMF : 1/EtOH : 20.

The preferred inorganic base is potassium carbonate.

10 The preparation of compounds of the invention will be more clearly illustrated by the following examples.

EXAMPLE 1

Preparation of N,N,N',N'-Tetraoctyl-ethylenediamine. (Tetocen).

In a 500 cm³ conical flask, 100g of octyl iodide is added to a 110 cm³ of 60/40 hexane/ethanol mixture containing 59,0g anhydrous
15 K₂CO₃. While this solution is being stirred 6,09g of ethylenediamine is added.

The flask and contents are placed on a heater stirrer and refluxed and stirred for 40 hours.

20 The solid unreacted K₂CO₃ and KI is filtered off and solvent removed on a rotary evaporator.

The remaining ditertiary amine (viscous oil) is taken up in an organic (water immiscible) solvent: petroleum ether or chloroform. HI is extracted into aqueous 3 mol dm⁻³ NaOH. The organic phase is then separated, washed with H₂O and dried over anhydrous Na₂SO₄. The solvent is
25 distilled off and the product distilled under high vacuum 0,05 mm Hg)

7.

at 180⁰-220⁰C in a 75% yield. Any unreacted octyl iodide comes over at 80-82⁰C.

The % free nitrogen base is calculated from a titration of the product in glacial acetic acid against 0,1 mol/dm³ HClO₄ soln. This assumed
5 the product to contain nitrogen in the form of tertiary nitrogen atoms. The results of C,H,N are in agreement with the stoichiometry of the ditertiary diamine viz.

Theoretical : C, 80,2%; H, 14,3%; N, 5,5%;

Found : C, 81,0%; H, 13,5%; N, 5,3%;

10 The infrared spectra indicated the absence of primary and quaternary N atoms.

EXAMPLE 2

Preparation of N',N',N',N,N,N-hexaoctyl hexamethylene=diammonium diiodide. (Hexochem)

15 53,0g of octyl iodide is placed in a 250 cm³ conical flask containing 19g anhydrous K₂CO₃, 18 cm³ ethanol and 0,5cm³ dimethylformamide. While this mixture is stirred 4,0g 1,6-diaminohexane is added.

The flask and contents are placed in a heater stirrer, refluxed and stirred for 70 hours.

20 The solid unreacted K₂CO₃ and KI is filtered off and the solvent is removed on a rotary evaporator.

The remaining viscous brownish oil is dissolved in chloroform and filtered to remove any K₂CO₃ or KI present.

The organic layer is shaken with a 3 mol dm⁻³ NaOH solution to remove
25 any hydro-iodide salts. These may be formed as a by-product as a result of insufficient stirring.

8.

The organic phase is separated, washed with H_2O and dried over anhydrous sodium sulphate.

The solvent is removed under reduced pressure never allowing the temperature to exceed $70^{\circ}C$.

- 5 The waxy solid is dried under a vacuum of 1 mm Hg, at $\approx 100^{\circ}C$ for 30 minutes and thus obtained in an 80% yield.

The % iodide is determined argentometrically by potentiometric titration.

EXAMPLE 3

- 10 Preparation of N,N'-Dimethyl-N,N',N'-tetra-octylethylene diammonium diiodide (Dimtetocen)
-

50g N,N,N',N'-tetraoctylethylenediamine (tetocen) and 40g methyl iodide are reacted by dissolving the tetocen in a minimum of chloroform and adding the methyl iodide to the solution.

- 15 The reactants are heated to $50^{\circ}C$ and refluxed for 40 hours with continuous stirring. Any unreacted methyl iodide and solvents are removed under reduced pressure. Care is taken not to heat to more than $80^{\circ}C$. The product is then dried under high vacuum (1mm Hg) at $100^{\circ}C$ for $\frac{1}{2}$ -hour. The yield is 90%.
- 20 In a similar manner, the compounds listed in Table I were prepared.

| Compound | R ₁ | R ₂ | R ₃ | n | X | Calculated | Found | Calculated | Found |
|---|----------------|----------------|----------------|---|----|------------|-------|------------|-------|
| $\begin{array}{c} R_1 \\ \diagup \\ N-(CH_2)_n-N \\ \diagdown \quad \diagup \\ R_2 \quad R_1 \\ \quad \quad R_2 \end{array}$ | octyl | octyl | | 2 | | 5,50 | 5,41 | | |
| | decyl | decyl | | 2 | | 4,51 | 4,38 | | |
| | *dodecyl | dodecyl | | 2 | | 3,82 | 3,51 | | |
| | octyl | octyl | | 3 | | 3,36 | 5,21 | | |
| | octyl | octyl | | 6 | | 4,96 | 4,81 | | |
| | dodecyl | dodecyl | | 6 | | 4,81 | 4,61 | | |
| $\left[\begin{array}{c} R_1 \\ \diagup \\ R_2-N-(CH_2)_n-N \\ \diagdown \quad \diagup \\ R_3 \quad R_3 \end{array} \right]_{2X}$ | octyl | octyl | methyl | 2 | I | | | 32,01 | 32,00 |
| | octyl | octyl | methyl | 2 | Cl | | | 11,62 | 11,55 |
| | dodecyl | dodecyl | methyl | 2 | I | | | 24,95 | 24,90 |
| | dodecyl | dodecyl | methyl | 2 | Cl | | | 8,50 | 8,31 |
| | heptyl | heptyl | heptyl | 5 | I | | | 26,41 | 26,39 |
| | heptyl | heptyl | heptyl | 6 | Cl | | | 9,11 | 8,99 |
| | octyl | octyl | octyl | 6 | I | | | 24,28 | 24,22 |
| | octyl | octyl | octyl | 6 | Cl | | | 8,22 | 8,17 |
| | | | | | | | | | |
| | | | | | | | | | |

*This product could not be purified by distillation.

10.

Further according to the invention, there is provided a process for extracting metal ions from an aqueous solution containing the ions in the form of anionic complexes, including the step of contacting the aqueous solution with an extractant of the type described above.

- 5 The ditertiary amine, phosphine and arsine extractants are suitable, in particular, for the extraction of cobalt, copper, zinc, silver, cadmium, the platinum group metals, uranium and other actinoids.

- The diquaternary ammonium, phosphonium and arsonium extractants are suitable, in particular, for the extraction of uranium and other
10 actinoids, cerium, vanadium, manganese, iron, cobalt, copper, zinc, silver, cadmium, tin, lead and the platinum group metals.

- The ditertiary amine, phosphine and arsine group of extractants can be used to extract metal ions in strongly acidic medium. Under these conditions the metal ion, as an anionic complex, is extracted into a
15 suitable organic phase by ion association with the protonated extractant. Examples will now be given.

EXAMPLE 4

- The extraction of UO_2SO_4 as a function of sulphuric acid concentration with Alamine 336, N,N,N',N'-tetraoctylhexamethylenediamine (tetochem),
20 N,N,N',N'-tetraoctyltrimethylenediamine (tetoctrim)
N,N,N',N'-tetraoctylethylenediamine (tetocen), into 15% isodecanol/benzene solution.

$0,44\text{g dm}^{-3}$ UO_2SO_4 is made up and standardised. The extracting organic phase is made up in isodecanol/benzene (5%) so that the organic phase is 7,10%, 6,09%, 5,22% and 5,08% with respect to Alamine 336, tectochem, tectoctrim and tetocen respectively.

Different solutions of H_2SO_4 are made up by diluting concentrated H_2SO_4 to give 1,82; 0,92; 0,46; 0,182; 0,092; 0,046; 0,0182; 0,0092 mol dm^{-3} solutions.

10 cm^3 of UO_2SO_4 solution and 10 ml H_2SO_4 (of each of the above) are pipetted into each of eight 50 cm^3 round bottom flasks. 20 cm^3 of organic solution is added to each flask. These are shaken for 15 min. to attain equilibrium.

The solutions are separated in 50 cm^3 separating funnels and the aqueous phase filtered (Whatman No. 40 filterpaper) and analysed.

The results obtained are given in Table III

TABLE III

| $\frac{\text{H}_2\text{SO}_4}{\text{mol dm}^{-3}}$ | Alamine | Tetocen | Tectoctrim | Tectochem |
|--|---------|---------|------------|-----------|
| 0,23 | 99,89 | 99,93 | 99,82 | 99,80 |
| 0,091 | 99,93 | 99,93 | 99,87 | 99,64 |
| 0,046 | 99,90 | 99,91 | 99,93 | 99,90 |
| 0,023 | 99,90 | 99,90 | 99,83 | 99,93 |
| 0,0091 | 99,21 | 99,52 | 99,60 | 99,62 |
| 0,0046 | 98,71 | 99,03 | 99,04 | 99,30 |

In the extraction of UO_2SO_4 the percentage extractions are also very high for Alamine, but in dilute sulphuric acid the dictaionic species have higher relative percentage extractions, however.

EXAMPLE 5Extraction of Fe(III) from HCl

A solution of $1,75 \text{ gdm}^{-3}$ Fe(III) is made by dissolving FeCl_3 (anhydrous) in water.

- 5 The organic extracting solution is made $0,17 \text{ mol dm}^{-3}$ and $0,085 \text{ mol dm}^{-3}$ for Alamine, and tectochem respectively.

Four HCl solutions are made to give 11,3 (conc HCl) 9,04; 6,78; 2,26 mol dm^{-3} .

10 10 cm^3 of the Fe(III) solution is mixed together with 10 cm^3 of each of the HCl solutions and 20 cm^3 of organic extracting solution. The mixture is shaken for 15 minutes and the aqueous phase separated and filtered. Fe(III) is determined by atomic absorption spectrophotometry.

The results are given in Table IV.

TABLE IV

| HCl | % extraction with Alamine | ppm left (Aqueous) | % extraction with N,N,N',N'-tetraoctyl-hexamethylene diamine | ppm left (Aqueous) |
|------|---------------------------|--------------------|--|--------------------|
| 5,65 | 99,78 | 2,0 | 99,91 | 0,8 |
| 4,52 | 99,70 | 2,7 | 99,87 | 1,2 |
| 4,07 | 99,48 | 4,7 | 99,42 | 5,3 |
| 1,13 | 98,77 | 11,2 | 98,71 | 11,7 |

EXAMPLE 6Extraction of Cu(II) from HCl

An aqueous solution of $4,00 \text{ dm}^{-3}$ Cu(II) (from CuCl_2) is made. HCl solutions are made up to give the required concentrations as set out in Table V.

10 cm^3 Cu(II) and 10 cm^3 HCl are mixed. 20 cm^3 of organic extracting phase containing Alamine and N,N,N',N'-tetraoctylethylenediamine are added. The mixture is shaken for 15 minutes and treated as in previous examples. Cu is analysed on an atomic absorption spectrophotometer. The results are given in Table V.

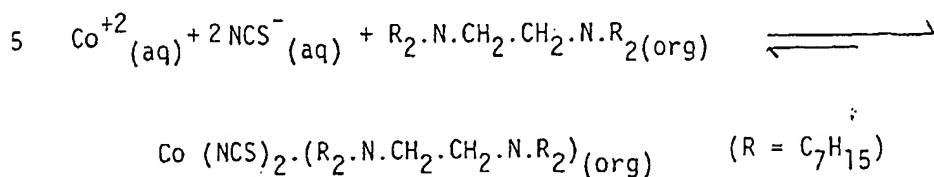
TABLE V

| HCl using mol dm^{-3} | % Cu extraction Alamine | % Cu extracted using tetocen |
|-----------------------------------|----------------------------|---------------------------------|
| 1,13 | 13,1 | 20,0 |
| 4,07 | 38,3 | 54,6 |
| 5,65 | 69,5 | 79,0 |
| 6,80 | 70,6 | 75,0 |
| 9,7 | 53,5 | 58,0 |

In a neutral or relatively alkaline solution the ditertiary amines, phosphines and arsines can act as neutral ligands and can complex with metal ions. In the presence of suitable anions the resultant complex can be extracted into the organic phase. An example thereof is now provided.

EXAMPLE 7

Cobalt, in the divalent form, can be extracted from dilute aqueous solutions in the presence of a small amount of thiocyanate into an organic phase. The reaction for the extraction is as follows:

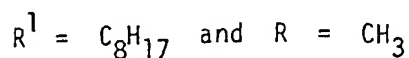
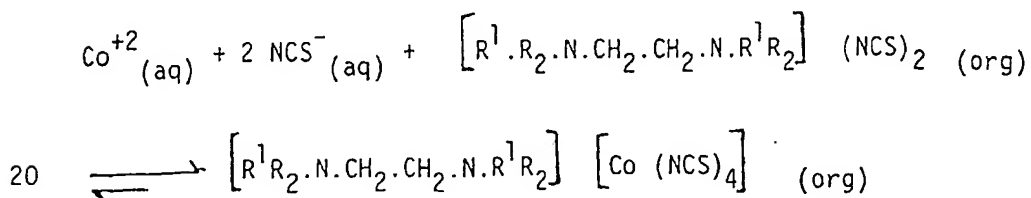


It is found that the organic phase can be stripped of cobalt by contacting it, with a slightly acidic aqueous solution. If the solution becomes more strongly acidic, extraction of the cobalt by the process described previously occurs.

The diquatery salt group of extractants can be used in the extraction of metal ions in both acidic and neutral solutions. The metal ion forms an anionic complex with anions from the solution and/or extractant which are then extracted into the organic phase by ion association with the cation of the extractant. Examples thereof are now given.

EXAMPLE 8

Cobalt, in the divalent form, can be extracted from dilute aqueous solution into an organic phase, as follows:



EXAMPLE 9Extraction of cobalt from nickel sulphate

Appropriate solutions of CoSO_4 , NiSO_4 and NH_4SCN are made up to give initial aqueous solution of 10 cm^3 containing $0,4012\text{ mmole Co(II)}$ and $40,12\text{ mmole of Ni(II)}$. 40 cm^3 organic extracting phase of $\text{N,N,N,N',N',N'-hexaoctylhexamethylenediamonium thiocyanate}$ (hexochem thiocyanate) is used and shaken with the aqueous. Similarly a ($0,0773\text{ mol dm}^{-3}$) $\text{N,N'-dimethyl-N,N,N',N'-tetraoctylethylenediamonium thiocyanate}$ organic solution is made up.

A second experiment involves the presence of NH_4SCN in ratios of $\text{Co} : \text{NCS} :: 1 : 5$.

RESULTS

TABLE VI(a) - NO NCS ADDED

| Extractant | % Co ext | ppm Co org | ppm Ni |
|---|----------|------------|--------|
| Aliquat | 2,39 | 56 | 0 |
| $\text{N,N'-dimethyl-N,N,N',N'-tetraoctylethylenediamonium thiocyanate}$ (dimtetocen) | 6,75 | 154 | 18 |
| Hexochem thiocyanate | 11,61 | 266 | 0 |

TABLE VI(b) - NCS ADDED TO AQUEOUS PHASE

| Extractant | NCS/Co | % Co ext | ppm Co org | ppm Ni |
|-------------|--------|----------|------------|--------|
| Aliquat 336 | 4,87 | 89,9 | 2049 | 16 |
| Dimtetocen | 4,87 | 61,0 | 1390 | 275 |
| Hexochem | 4,97 | 88,8 | 2025 | 2,6 |

The extractant Hexochem has a high specificity for cobalt and 99% extraction of cobalt from a cobalt solution in the presence of a hundredfold excess nickel has been achieved on a single contacting.

5 The efficiency of the compounds of this invention can be enhanced by using them in combination with organic solvents with low dielectric constants which favour ion pair extraction. Suitable solvents include benzene, solvesso hexane and their mixtures with long chain alcohols.

10 A further example illustrating the use of the compounds in extracting metal ions from solution and providing comparative data with other organic solvent extractants is given below.

EXAMPLE 10

15 An organic extracting phase is made up (10 mol dm^{-3}) by dissolving 7,33g N,N,N',N'-tetradodecylethylenediamine(tetdodecen) in 5% (v/v) isodecanol/benzene mixture. This is made up to the mark in a 100 cm^3 volumetric flask, with the benzene solution. The concentration of tetdodecen in the mixture is checked by free nitrogen titration against standard HClO_4 in non-aqueous solvent, e.g. acetic acid.

20 An aqueous solution of UO_2Cl_2 (6.4 g/l) is made by dissolving $\text{UO}_2\text{Cl}_2(\text{H}_2\text{O})_3$ in water. Six solutions of HCl are prepared having the following concentrations: 11,30; 9,07; 8,13; 6,78; 2,26; mol dm^{-3} .

25 5 cm^3 of UO_2Cl_2 is mixed with 15 cm^3 of HCl solutions, to give $[\text{HCl}] = 8,48; 6,78; 6,16; 5,09; 4,24$ and $1,70 \text{ mol/dm}^3$. 20 cm^3 of organic phase is added to each and the mixtures are shaken for 15 minutes to allow equilibrium to be attained. The two phases are poured into a separating funnel and the aqueous phase passed through a filter (Whatman No. 40). The aqueous phase is analysed for uranium by a potentiometric method..

The following results are thus obtained and compared to extraction data obtained using Alamine 336 (an industrially used amine). The Alamine 336 concentration used is double, in terms of mol dm^{-3} , that of the tetradecan used to obtain the same number of nitrogen centres.

5

| TABLE II | | | | |
|----------|--------------|---------|----------|---------|
| HCl | % Extraction | | | |
| | Tetradecan | Alamine | Hexochem | Aliquat |
| 8,48 | 98,4 | 94,3 | 97,9 | 98,4 |
| 6,78 | 98,1 | 91,2 | 98,4 | 98,9 |
| 6,10 | 97,0 | 86,9 | 99,9 | 98,7 |
| 5,09 | 93,3 | 73,0 | 99,5 | 98,5 |
| 4,24 | 83,4 | 58,5 | 98,7 | 98,2 |
| 1,70 | 19,1 | 4,1 | | |

Hexochem = N,N,N,N',N',N'-hexaoctylhexamethylenediamine

If the maximum extraction efficiencies of the two pairs are compared then the dicationic extractant which shows the least improvement when compared to its monocationic analogue, leaves only 0,1% of the metal in the aqueous phase as compared to the 1,3% of its mono analogue i.e. a tenfold difference.

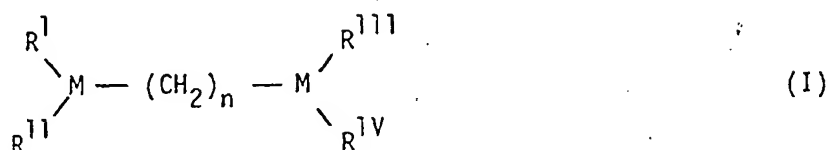
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CLAIMS:

1.

A compound selected from:

- 5 (a) a ditertiarydiamine, diphosphine or diarsine of the general formula

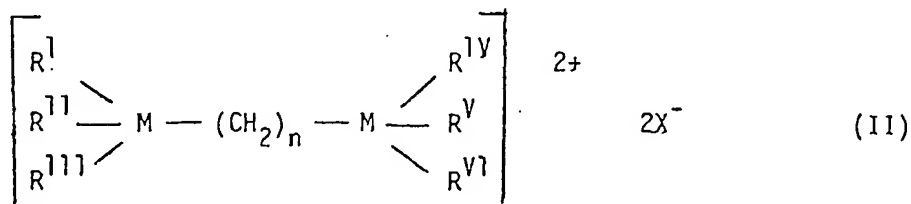


where M = N, P or As

n = 2 to 10 inclusive

- 10 R^I , R^{II} , R^{III} , and R^{IV} are the same or different and each represents an alkyl or an aryl group having 7 to 12 carbon atoms; and

- (b) a diquaternary diammonium, diphosphonium or diarsonium salt of the general formula



where M = N, P or As

n = 2 to 10 inclusive

- 15 $X = Cl^-, Br^-, I^-, SCN^-, NO_3^-, HSO_4^-$ or $SO_4^{=}$,

R^I , R^{II} , R^{III} , R^{IV} , R^V , and R^{VI} are the same or different and each represents an alkyl or an aryl group having 1 to 12 carbon atoms or H, provided that at least four of R^I , R^{II} , R^{III} , R^{IV} , R^V , and R^{VI} represent an alkyl or an aryl group having 7-12 carbon atoms.

19.

2.

A compound according to claim 1 wherein the substituents on one M atom are the same as the substituents on the other M atom, M being as defined in claim 1.

3.

5 A compound of Formula (1) according to claim 1 wherein $M=N$, $n=2$, and $R^I=R^{II}=R^{III}=R^{IV}$ =octyl or decyl or dodecyl.

4.

A compound of Formula (1) according to claim 1 wherein $M=N$, $n=3$ and $R^I=R^{II}=R^{III}=R^{IV}$ =octyl.

5.

A compound of Formula (1) according to claim 1 wherein $M=N$, $n=6$, and $R^I=R^{II}=R^{III}=R^{IV}$ =octyl or dodecyl.

6.

10 A compound of Formula (11) according to claim 1 wherein $M=N$, $n=2$, $X=I$ or Cl , $R^I=R^{II}=R^{III}=R^{IV}$ =octyl or dodecyl, and $R^V=R^{VI}$ =methyl.

7.

A compound of Formula (11) according to claim 1 wherein $M=N$, $n=6$, $X=I$ or Cl , $R^I=R^{II}=R^{III}=R^{IV}=R^V=R^{VI}$ =heptyl or octyl.

8.

A compound of claim 1 which is N,N,N',N' ,-tetraoctylethylene diamine.

9.

15 A compound of claim 1 which is N',N',N',N,N,N ,-hexaoctylhexamethylene diammonium diiodide.

10.

A compound of claim 1 which is N,N' ,-dimethyl- N,N,N',N' ,-tetraoctylethylene diammonium diiodide.

11.

A compound according to claim 1 for use in the solvent extraction of metal ions from an aqueous solution containing the ions in the form of anionic complexes.

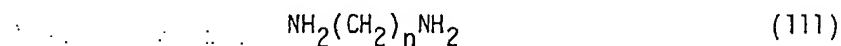
12.

5 A compound according to any one of claims 2 to 10 for use in the solvent extraction of metal ions from an aqueous solution containing the ions in the form of anionic complexes.

13.

A process for the production of the compounds of Formula (I) of claim 1 in which M=N comprises the steps of directly alkylating a compound of the Formula (III)

10



in a suitable alcoholic solvent mixture in the presence of an inorganic base to give a compound of the Formula (Ia)



15 wherein R represents an alkyl or an aryl group having 7 to 12 carbon atoms and n is as defined in claim 1.

14.

A process according to claim 13 wherein the alcoholic solvent mixture is a mixture of ethanol and hexane.

15.

A process according to claim 14 wherein the ratio of ethanol to hexane is ethanol:40/hexane:60 by volume.

16.

20 A process according to any one of claims 13 - 15 wherein the inorganic base is a carbonate.

17.

A process according to claim 16 wherein the carbonate is potassium carbonate.

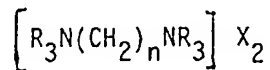
21.

18.

A process for the production of the compounds of the Formula (11) of claim 1 in which M=N comprises the steps of directly alkylating a compound of the formula (111)



5 in a suitable alcoholic solvent mixture in the presence of an inorganic base to give a compound of the formula (11a)



wherein R represents an alkyl or an aryl group having 7 to 12 carbon atoms, and n and X are as defined in claim 1.

19.

10. A process according to claim 18 wherein the alcoholic solvent mixture is a mixture of ethanol and DMF.

20.

A process according to claim 19 wherein the ratio of ethanol to DMF is DMF:1/ethanol:20 by volume.

21.

15 A process according to any one of claims 18 to 20 wherein the inorganic base is a carbonate.

22.

A process according to claim 19 wherein the carbonate is potassium carbonate.

22.

23.

A process for extracting metal ions from an aqueous solution containing the ions in the form of anionic complexes includes the steps of contacting the aqueous solution with a compound according to any one of claims 1 to 10.

24.

- 5 A process according to claim 23 wherein the compounds of Formula (1) of claim 1 are used for the extraction of ions of cobalt, copper, zinc, silver, cadmium, the platinum group metals, uranium and the other actinoids.

25.

- 10 A process according to claim 23 wherein the compounds of Formula (1) of claim 1 are used for the extraction of ions of uranium and the other actinoids, cerium, vanadium, manganese, iron, cobalt, copper, zinc, silver, cadmium, tin, lead and the platinum group metals.

26.

A process according to claim 23 wherein the compounds of Formula (1) of claim 1 are used to extract metal ions in strongly acidic medium.

27.

- 15 A process according to claim 23 wherein the compounds of Formula (1) of claim 1 are used to extract metal ions from neutral or alkaline solutions by complexing the metal ions with a compound of Formula (1) and extracting the resultant complex into the organic phase.

28.

- 20 A process according to claim 27 wherein the metal ion is removed from the organic phase by contacting it with a slightly acidic aqueous solution.

29.

A process according to claim 23 wherein the compounds of Formula (1) of claim 1 are used for the extraction of metal ions from acidic and neutral solutions.

23.

30.

A process according to any one of claims 23 to 29 wherein the compounds of claim 1 are used in combination with organic solvents of low dielectric constants.

31.

5 A process according to claim 30 wherein the solvent of low dielectric constant is selected from the group of benzene, solvesso hexane, and mixtures thereof with long chain alcohols.

32.

A compound according to claim 1, other than the compounds of claims 8 to 10, substantially as herein specifically described with reference to the Examples.

33.

10 A process according to claim 13 substantially as herein described with reference to Example 1.

34.

A process according to claim 18 substantially as herein described with reference to either of Examples 2 or 3.

35.

15 A process according to claim 23 substantially as herein described with reference to any one of Examples 4-10.

DATED THIS 12TH DAY OF NOVEMBER, 1980.


SPOOR AND FISHER

APPLICANT'S PATENT ATTORNEYS